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(71) Applicant: BENCKISER CONSUMER PRODUCTS, INC. [US/US]; Greenwich American Centre, Five American Lane, Greenwich, CT 06831-2561 (US).

(72) Inventors: PIROLO, Robert, S.; 1144 North Vassault, Tacoma, WA 98406 (US). CLARK, Allen, M.; 5606 W. Old Stump Drive, Gig Harbor, WA 98332 (US).

(74) Agent: KALIS, Janal, M.; Oppenheimer Wolff & Donnelly LLP, Suite 3400, 45 South Seventh Street, Minneapolis, MN 55402 (US).

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(54) Title: SANITIZING ANTIMICROBIAL RINSE AID

(57) Abstract

The present invention includes a method for killing microorganisms on an article that is rinsed in water at a temperature that is less than 140 °F. The method includes a step of treating the rinse water with an antimicrobial rinse aid. The rinse aid is comprised of a nonionic surfactant. The rinse aid is also comprised of a quaternary ammonium compound and a solubilizing agent. The antimicrobial rinse aid treated dish water is exposed to the articles. The rinse water is heated after exposure to a temperature that is less than 140 °F.

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SANITIZING ANTIMICROBIAL RINSE AID

BACKGROUND OF THE INVENTION

The need for a sanitizing antimicrobial rinse aid for automatic dishware washers is increasing with the emergence of new food borne pathogens. This increase is documented by reports from state health departments and from the CDC. Outbreaks of Salmonella enteritidis, E. coli 0157:H7, Campylobacter, H. pylorii, Staphylococcus aureus, and Enterococci, are all being reported in increasing numbers. A five fold increase in diseases caused by Salmonella enteritidis is expected in the next decade by the CDC.

Automatic consumer or household dishwashers now may fail to sanitize plates, glasses and flatware, because the prolonged high temperatures required for sanitation are not obtained in current automatic household dishwashers. This occurs for two reasons: 1) the water entering the dishwasher rarely exceeds 120° F., in order to prevent scalding and to conserve energy, 2) the dishwasher temperature cycle is not high enough, over 140° F., for a long enough time, over 30 minutes, to kill pathogenic bacteria. Our studies have shown the persistence of bacteria in household automatic dishwashers 30 minutes after the end of the wash cycle. With the increased contamination of food and of food preparation sites by disease causing bacteria such as E. coli 0157:H7 and Salmonella species, there is a need for a chemical sanitizer in the final rinse in household automatic dishwashers. If used in commercial dishwashers there would be a significant economic advantage due to energy savings and decrease in energy expenditures.

Other patented rinse aid sanitizing chemicals, such as chlorine have been used. U.S. Patent 5,358,653 Galdfelter et al. teaches the use of a solid system containing a highly active available chlorine source and sheeting agents. The system must be solid to permit manufacture, storage, distribution, sale, and consumption of the rinse aid, and to prevent chemical degradation of the chlorine source. An aqueous liquid system would be better for daily to monthly use in the rinse aid dispenser built in to most household automatic dishwashers.

Water used in the rinse cycle of automatic dishwashing machine frequently has substantial hardness component consisting of calcium and magnesium ions, which in the presence of certain final rinse material can precipitate and leave unsightly deposits of mixed calcium and magnesium salts. These deposits can also contain other hardness components

such as ferrous or ferric compounds and other common cations. There is a need to incorporate sequestrants into the formulas to overcome this problem.

SUMMARY OF THE INVENTION

The present invention includes a method for killing microorganisms on an article that is washed in water at a temperature that is less than 140° F. and that is rinsed in rinse water. The method includes a step of treating the rinse water with an antimicrobial rinse aid. The rinse aid is comprised of a surfactant with low to no foaming properties. The rinse aid is also comprised of a quaternary ammonium compound and a solubilizing agent. The antimicrobial rinse aid treated dishwater is exposed to the article. The wash water is heated either before exposure, after exposure or during exposure to a temperature of less than 140° F.

The present invention also includes an antimicrobial rinse aid. The rinse aid is comprised of a nonionic surfactant selected from the group consisting of a fatty alcohol C_{12-14} comprised of three ethylene oxide groups and six propylene oxide groups; a block polymer of ethylene oxide and propylene oxide; a low foaming mixture of nonophenol surfactants or an alkoxylated linear alcohol. The rinse aid also includes a quaternary ammonium compound and a solubilizing agent such as lactic acid.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

To create an aqueous liquid antimicrobial rinse aid several components are necessary, a surfactant with low foaming properties, an antimicrobial agent, and a solubilizing agent to aid in the maintenance of a clear and stable solution. It is preferred that the surfactant be nonionic to avoid chemical reactions with other components. The antimicrobial agent should meet the criteria of being broad spectrum, short acting, and food safe. The solubilizing agent should be compatible with the other components, capable of complexing metallic cations, and if possible synergistic to the antimicrobial agent.

Review of various low foam nonionic surfactants available today, led us to evaluate the four surfactants listed below, as examples of such surfactants:

C_{12-14}, 3 ethylene oxide (EO) (CH_2-CH_2-O), 6 propylene oxide groups (CH_2-CH-$_3$-CH-O)

Henkel Dehypon® LS 36, a fatty alcohol C_{12-14}, 3 EO, 6 PO

BASF Industrol® N-3, a block copolymer EO PO

Union Carbide Triton® Nonionic Surfactant CF 32, a low foaming mixture of nonophenol surfactants

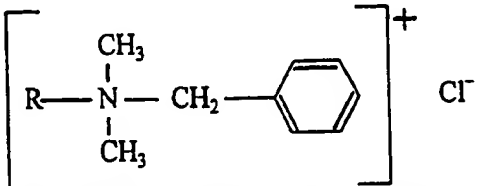
Olin Poly-Tergent® SLF 18, an alkoxylated linear alcohol

The BASF Industrol® N-3 is manufactured by the BASF Corporation in Mount Olive, New Jersey. The Industrol® N-3 is a nonionic surfactant which is designed to provide the combined performance requirement of sheeting and low foaming under variable field conditions. The active agent in the Industrol® surfactant is polyoxypropylene polyoxyethylene block copolymer. The viscosity of this nonionic surfactant falls within a range of 713-755 centipoise at 25° C.

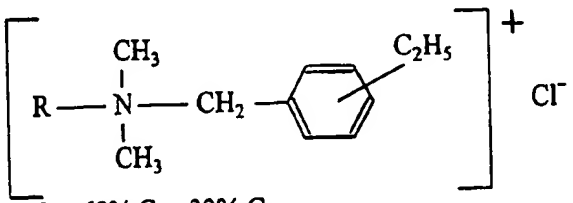
The Olin Poly-Tergent® SLF 18 material is manufactured by Olin Corporation of Norwalk, Connecticut. This Poly-Tergent® material is comprised of poly(oxyethylene/ polyoxypropylene) monohexylether. The Poly-Tergent® material has a freezing point of 0°-8° C. and a decomposition temperature in a range of 204°-218° C. The material has a pH at 25° C. of 5-7 as a 1% aqueous solution.

The Union Carbide Triton® Nonionic Surfactant CF 32 is manufactured by Union Carbide in South Charleston, West Virginia. This surfactant has a pH of 9.5-11 in a 5% aqueous solution. This surfactant has a cloud point of 1% in an aqueous solution of 20°-27° C. The surfactant formulation includes a water concentration of 5.0%-6.5% by weight.

The antimicrobial agent, should be short acting, and accepted for food use. Therefore, we chose a quaternary ammonium compound, such as Stepan 2125 M Quat, because it was available in an 80% active aqueous solution. The Stepan 2125 M Quat is also known as BTC 2125® M product. It is manufactured by Stepan Company in Northfield, Illinois. The Stepan 2125 M Quat is comprised of an N-alkyl dimethyl benzyl ammonium chloride and N-alkyl dimethyl ethyl benzyl ammonium chloride as is shown in the chemical formulations described below:



R = 60% C_{14}, 30% C_{16}, 5% C_{12}, 5% C_{18}



R = 68% C_{12}, 32% C_{14}

In particular, the active ingredients of the 2125 M quaternary ammonium product include N-alkyl (60% C_{14}, 30% C_{10}, 5% C_{12}, 5% C_{18}) dimethyl benzyl ammonium chloride in a concentration of 25% by weight. The 2125 M quaternary ammonium product further

includes N-alkyl (68% C_{12}, 32% C_{14}) dimethyl ethyl benzyl ammonium chloride in a concentration of 25%. Inert ingredients comprise 50% of the 2125 M preparation. The pH of a 10% aqueous solution of the 2125 M product is 6.5-8.5. The viscosity of the 2125 M Quat product is 50 centipoise at 25° C. The material has a pour point of -4° C. and a flash point of 77° C. The 2125 M Quat product is biodegradable.

The solubilizing agent, and sequestrant, should be a weak organic acid with multiple reactive groups to allow a buffering effect and to have metal sequestering properties. A preferred embodiment could include lactic acid, or other similar acids such as citric acid. One lactic acid source is 88% lactic acid from ADM, Decatur, Illinois.

The solutions were prepared by taking the solutions of the surfactants, the solution of the quaternary ammonium compound and the solution of the sequestrant, all obtained from the manufacturer, and mixing the solutions to form a clear solution. The percentages expressed by weight are indicated by the general formula Quat 50%, Surfactant 40% and Sequestrant 10%, i.c. 50 grams of 80% Stepan 2125 M was weighed into a beaker, 40 grams of Henkel surfactant solution was added, followed by 10 grams of 88% lactic acid solution, with the total weight being 100 grams.

Final Dilution of Antimicrobial Rinse Aid

In order to test if the formulas could achieve a working final concentration of quaternary ammonium compound the following calculations were madc:

We initially calculated the amount needed in a concentrate, and then demonstrated the effectiveness of the diluted material in laboratory experiments.

Average rinse volume of dishwashers was estimated to be 1.84 U.S. gallons, or 6947.84 ml based on information obtained from five manufacturers representing 90% of the machines marketed in the United States. The amount in gallons was converted to milliliters:

$$1.84 \text{ gal} \times 128 \text{ oz/gal} \times 29.5 \text{ml/oz} = 6947.84 \text{ ml}$$

The usual amount of rinse aid dispensed in rinse cycle is three ml, 3.0 ml, according to the manufacturers. With 40% quaternary ammonium compound in Antibacterial Rinse Aid Formulas of the present invention, a level of between 150 and 200 ppm can be achieved in the rinse phase of the usual automatic dishwasher in use in the United States.

3.0 ml/6947.84 ml x 40% x 10000 ppm/1% = 172.7 ppm

Therefore we concluded that it was possible to make a working formula and test it for antimicrobial activity.

EXAMPLES

Demonstration of Antimicrobial Activity

Two standard microbiologic techniques were used to demonstrate the antimicrobial effect of the rinse aid formulas of the present invention compared to quaternary ammonium compound diluted in deionized water.

1. A modified disk antimicrobial diffusion method of Bauer et al. was used on lawn plate cultures of specific bacteria found in dishwashers and on similar lawn cultures of specified common food pathogens. Cultures were inoculated with 50 µl of diluted agent and observed for antimicrobial effect at 48 hours. Antimicrobial effect is shown by areas of clearing on the culture plates.
2. American Association of Analytical Chemists, AOAC, in use dilutions of four rinse aid formulas were performed using a 30 second exposure of a Gram positive organism, a Gram negative organism, and a yeast. After the 30 second exposure, the quaternary compound was neutralized and quantitativc recovery of organisms remaining was performed by tube dilution and quantitative transfer of 50 µl of diluted bacteria to growth plates. Plates were incubated at 35° C. and observed at 24 hours. Remaining microorganisms were counted and recorded. See results below.

Results

The results of the minimum inhibitory concentration experiments are shown in Table I. All of the Antimicrobial Rinse Aid Formulas of the present invention were active against all of the microorganisms used in the test.

1. Henkel LS 36, Stepan 2125 M Quat, Lactic Acid
2. BASF N-3, Stepan 2125 M Quat, Lactic Acid
3. Union Carbide CF 32, Stepan 2125 M Quat, Lactic Acid
4. Olin SLF 18, Stepan 2125 M Quat, Lactic Acid
5. Stepan 2125 M Quat, no surfactant, no acid.

General Antimicrobial Rinse Aid formula: Quat 50% (80% active), Surfactant 40%, Lactic Acid 10%, all by weight.

TABLE I

Minimum Inhibitory Concentrations (ppm) of Four Rinse Aid Formulas Compared to Quaternary Ammonium Compound Alone.

Organism/ Rinse Aid	1 Henkel	2 BASF	3 Union Carbide	4 Olin	5 Stepan Quat
S. aureus	<50	<50	<50	<50	<50
E. coli	200	<50	<50	<50	<50
S. enter.	<50	<50	<50	<50	<50
Shigella	<50	<50	<50	<50	<50
C. albicans	<50	<50	<50	<50	<50

The results of the In Use Dilution Test (AOAC) are shown in Table II. All of the Antimicrobial Rinse Aid Formulas of the present invention reduced the bacterial count by more than 99.999% (>99.999) when tested against the bacteria listed below grown in liquid suspension. A Gram positive organism, Staphylococcus aureus, S. aureus; a Gram negative organism, Escherichia coli, E. coli; and a yeast, Candida albicans, C. albicans, were chosen as the test organisms.

TABLE II

In Use Dilution Test (AOAC) of Five Rinse Aid Formulas
(Percentage Reduction of Bacteria and Yeast in Liquid by Quat at 100 ppm)

Organism/ Rinse Aid	Conc. of Organism	1 Henkel	2 BASF	3 Union Carbide	4 Olin	5 Stepan Quat
S. aureus	$6.8x10^8$	>99.999	>99.999	>99.999	>99.999	>99.999
E. coli	$1.5x10^9$	99.733	>99.999	99.867	>99.999	>99.999
C. albicans	$1.6x10^9$	99.575	99.995	78.75	>99.999	>99.999

Conclusion

The above two test methods used above demonstrate the antimicrobial effect of the four rinse aid formulas by both minimal inhibitory concentration method on lawn plate cultures of selected microorganisms and by the in use dilution method recommended by the AOAC. All of the rinse aid formulas were effective against the microorganisms in the minimum inhibitory concentration test on lawn cultures. The formula containing Henkel surfactant did require a 200 ppm level to inhibit the growth of E. coli. In the AOAC use dilution test most formulas were effective against Gram positive and Gram negative organisms. As can be seen from Table II, the only surfactant system, when used in formulation, to produce a five log reduction in C. albicans was the alkoxylated linear alcohol manufactured by Olin. Therefore we chose this surfactant as the preferred embodiment.

Demonstration of Decreased Spotting of Dishware

In order to evaluate the rinse aid's antispotting and antifilming properties of the antimicrobial rinse aid formulas, the following protocol was used. Dishware: glass, pottery, and metallic tableware were processed through the wash and rinse cycle of a household mechanical automatic dishwasher, with and without the use of the various sanitizing antimicrobial rinse aid formulas for 10 cycles. The water was moderately hard. Automatic dishwashing soap was used in the amount recommended by the manufacturer. The amount of spotting, and filming of dishware was evaluated by trained observers using the following criteria and scale:

Feature/ Score	0	1	2	3	4
Spots	none	1-2	3-4	5-7	8-12
Film	none	slight	grey	grey/white	white

The results of the test of antispotting and antifilming functions are shown in Table III.

TABLE III

Results of Four Rinse Aid Formulas and Water on Spot Formation in an Automatic Dishwasher

Rinse Aid/ Features	1 Henkel	2 BASF	3 Union Carbide	4 Olin	5 Water/No Rinse Aid
Spots	0	0	1	1	3
Film	2	2	0	0	2

Conclusion

Each of the four rinse aid formulas showed less spotting and filming than the cycles using water alone.

IN THE CLAIMS

What is claimed is:

1. A rinse aid composition for rinsing dishware in an automatic dishwasher comprising:
a low to no foaming surfactant characterized in that it further comprises a water-insoluble quaternary ammonium compound with antimicrobial activity.

2. A method of rinsing dish water with rinse water and disinfecting dishware comprising:
treating the rinse water with a rinse aid comprising a low foaming surfactant;
contacting the rinse aid treated rinse water to the article; and
heating the rinse water to a temperature of less than 70°C characterized in that the rinse aid comprises a water-insoluble quaternary ammonium compound with antimicrobial activity.

3. A method for killing microorganisms on an article washed in water at a temperature of less than 70° C. and mixed in rinse water, comprising:
treating the rinse water with an antimicrobial rinse aid comprising a low to no foaming nonionic surfactant, a quaternary ammonium compound and a solubilizing agent; and
contacting the antimicrobial rinse aid treated rinse water to the article; and
heating the wash water to a temperature less than 70° C.

4. The method of claim 3 wherein the low to no foaming surfactant is selected from a group consisting of:
a fatty alcohol, C_{12-14}, with three ethylene oxide groups and six propylene oxide groups;
a block copolymer with at least 2 ethylene oxide groups and 2 propylene oxide groups;
a low foaming mixture of nonophenol surfactant; or
an alkoxylated linear alcohol.

5. The method of claim 3 wherein the quaternary ammonium compound is an 80% active aqueous ammonium solution.

6. The method of claim 3 wherein the solubilizing agent comprises lactic acid.

7. The method of claim 3 wherein the solubilizing agent comprises citric acid.

8. An antimicrobial rinse aid, comprising:
a low foaming surfactant selected from the group consisting of C_{12-14}, three ethylene oxide groups and six propylene oxide groups, a fatty alcohol C_{12-14}, comprised of three ethylene oxide groups and six propylene oxide groups;
a block copolymer of ethylene oxide and propylene oxide;
a low-foaming mixture of nonophenol surfactants; or
an alkoxylated linear alcohol;
a quaternary ammonium compound; and
a solubilizing agent such as lactic acid.

9. The agent of claim 8 wherein the quaternary ammonium compound is present in a concentration of 50% by weight.

10. The agent of claim 9 wherein the surfactant is present in a concentration of 40% by weight.

11. The agent of claim 10 wherein the sequestrant is present in a concentration of 10% by weight.

12. The agent of claim 8 wherein the quaternary ammonium compound is comprised of one or more of N-alkyl dimethyl benzyl ammonium chloride or N-alkyl dimethyl ethyl benzyl ammonium chloride.

13. The agent of claim 8 wherein the surfactant is comprised of polyoxypropylene-polyoxyethylene block copolymer.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 98/06535

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C11D1/835 C11D3/48

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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[X] Further documents are listed in the continuation of box C.

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Date of the actual completion of the international search	Date of mailing of the international search report
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